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Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF TIN AND LEAD IN SECONDARY TIN

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

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Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF TIN AND LEAD IN SECONDARY TIN

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Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF TIN AND LEAD IN SECONDARY TIN

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 February 1972, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 This standard prescribes methods for the determination of tin and lead in secondary tin in the ranges as specified in IS : 4280-1967*. For the methods for determinations of antimony, arsenic, copper, iron and bismuth in secondary tin, reference shall be made to IS : 1940-1969†.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and the practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from '1970 Annual Book of ASTM Standard : Part 32 Methods for chemical analysis of metals' issued by the American Society for Testing and Materials.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960‡.

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1. SCOPE

1.1 This standard prescribes the methods for determination of tin (96 to 99 percent) and lead (0·9 to 3·5 percent) respectively in secondary tin (*see* IS : 4280-1967*).

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with the procedure laid down in IS : 1817-1961§.

*Specification for refined secondary tin.
†Methods of chemical analysis of tin ingot (*first revision*).
‡Rules for rounding off numerical values (*revised*).
§Methods of sampling non-ferrous metals for chemical analysis.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1961*) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF TIN BY THE IODIMETRIC METHOD

4.1 Outline of the Method — Tin, in solution of the sample, is reduced in hydrochloric acid medium by antimony and aluminium and the reduced tin is titrated with standard potassium iodate solution using starch as indicator.

4.2 Reagents

4.2.1 *Concentrated Sulphuric Acid* — sp gr 1·84 (conforming to IS : 266-1961†).

4.2.2 *Potassium Bisulphate* — solid.

4.2.3 *Concentrated Hydrochloric Acid* — sp gr 1·16 (conforming to IS : 265-1962‡).

4.2.4 *Antimony Trichloride Solution* (3·2 *g*/100 *ml*) — Dissolve 0·80 g of antimony trichloride in 25 ml of concentrated hydrochloric acid.

4.2.5 *Aluminium Metal Drillings* — pure.

4.2.6 *Sodium Bicarbonate Solution* — saturated.

4.2.7 *Starch Solution* (10 *g*/*l*) — Make a paste of one gram of soluble starch in about 5 ml of water. To this add 100 ml of boiling water. Cool, add 5 g of potassium iodide (KI), and stir until KI is dissolved. Prepare fresh solution as needed.

4.2.8 *Standard Potassium Iodate Solution* (0·1 *N*) — Twice recrystallize potassium iodate from water and dry at 180°C to constant weight. Dissolve 3·567 g of potassium iodate in 200 ml of water containing one gram of sodium hydroxide and add 10 g of potassium iodide. When the solution is complete, dilute to one litre in a volumetric flask. Standardize the solution against pure standard tin solution (**4.2.9**) following the procedure described under **4.3.2** and **4.3.3**.

4.2.9 *Standard Tin Solution* (1 *ml* = 1·0 *mg Sn*) — Transfer 1·0000 g of tin (purity 99·85 percent, *Min*) to a 400-ml beaker and cover. Add 300 ml of dilute hydrochloric acid (1:1) and warm gently until the metal is dissolved. If dissolution is difficult, add 0·5 to 1·0 g of potassium chlorate ($KClO_3$). Cool, transfer to a 1-litre volumetric flask, dilute to volume, and mix.

*Specification for water, distilled quality (*revised*).
†Specification for sulphuric acid (*revised*).
‡Specification for hydrochloric acid (*revised*).

4.3 Procedure

4.3.1 Take 0·20 g of an accurately weighed sample in a 500-ml conical flask. Add 20 ml of concentrated sulphuric acid and 5 g of potassium bisulphate. Heat slowly to decompose the sample avoiding too high a temperature during initial heating. Then heat vigorously over an open flame till copious fumes of sulphur trioxide are evolved. Care shall be taken to remove the sulphur from the top of the flask.

4.3.2 Cool, add 150 ml water and 60 ml of concentrated hydrochloric acid. Add 1 to 2 drops of antimony trichloride solution and 2 g of aluminium metal drillings little at a time. After the last addition, stopper the flask quickly (*see* Fig. 1). Boil the solution with continuous evolution of gas for about 10 to 15 minutes until all the aluminium and metallic tin is in solution.

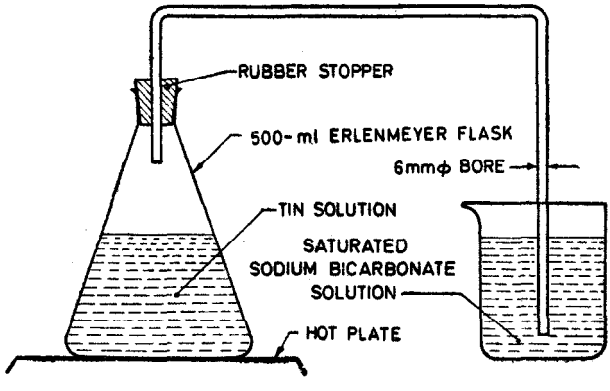

FIG. 1 APPARATUS FOR REDUCTION OF TIN

4.3.3 After the reduction is complete, dip the end of the siphon into a saturated solution of sodium bicarbonate and set aside to cool to about 25°C. When cool, remove the stopper, add 5 ml of starch solution and titrate the solution with standard potassium iodate solution.

4.4 Calculation

$$\text{Tin, percent} = \frac{A \times B}{C} \times 100$$

where

A = volume in ml of the standard potassium iodate solution required for the sample,

B = tin equivalent of the standard potassium iodate solution in g/ml, and

C = mass in g of the sample taken.

5. DETERMINATION OF LEAD BY THE SULPHATE METHOD

5.1 Outline of the Method — After removal of tin and antimony as volatile bromides, lead is precipitated as lead sulphate, dried and weighed.

5.2 Reagents

5.2.1 *Hydrobromic Acid-Bromine Mixture* — Mix 20 ml of bromine and 180 ml of hydrobromic acid.

5.2.2 *Perchloric Acid* — 70 percent.

5.2.3 *Lead Acid Solution* — Dissolve 0·5 g of lead nitrate in 200 ml of water and add with stirring 5 ml of concentrated sulphuric acid (sp gr 1·84) to the solution. Allow to stand for 24 hours and siphon or decant through a fine filter paper. Discard the precipitate.

5.2.4 *Ethyl Alcohol* — 95 percent.

5.3 Procedure

5.3.1 Take 0·5 to 2 g sample in a 250-ml wide-mouth flask. Add 30 ml of hydrobromic acid-bromine mixture and heat gently until the dissolution of the sample is complete avoiding excessive loss of bromine. Ensure complete removal of tin by slow evaporation, if necessary by repeated additions of hydrobromic acid-bromine mixture. Add 10 ml of perchloric acid and heat over an open flame until white fumes first appear. Continue heating moderately to decompose lead bromide and to expel all hydrobromic acid taking care to see that the excessive loss of perchloric acid does not take place.

5.3.2 If the solution is not clear, add 10 ml hydrobromic acid-bromine mixture, again heat to expel and to decompose lead bromide. Repeat

the process several times until a clear solution is obtained. Finally add 50 ml of lead acid solution and continue heating until the volume of the solution is reduced to 8 ml. Cool and dilute with water to 50 ml.

5.3.3 Boil the solution gently to dissolve the soluble salts. Cool and allow to stand at 50°C for 1 to 2 hours. Cool to room temperature and filter through sintered glass crucible of porosity No. 4. Wash the precipitate with cold lead acid solution and finally with ethyl alcohol. Dry the precipitate at about 120°C to constant mass.

5.4 Calculation

$$\text{Lead, percent} = \frac{A \times 68{\cdot}33}{B}$$

where

A = mass in g of lead sulphate, and

B = mass in g of the sample used.

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